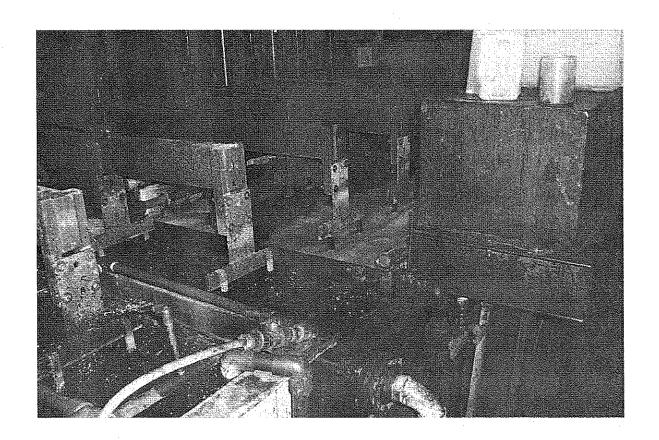


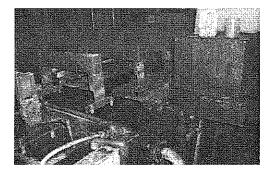
# **PFOS CHROMIUM ELECTROPLATER STUDY** U.S. ENVIRONMENTAL PROTECTION AGENCY-REGION 5



September 2009 **Final Report** 

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Cover: View of Chromium Electroplating Line

Photo: Mark Conti & David Barna U.S. EPA-Region 5-Cleveland Office Acronyms

Minnesota Mining, and Manufacturing Corporation

ANPRM Advance Notice of Proposed Rulemaking

CAA Clean Air Act

CARB California Air Resources Board

Cr(VI) hexavalent chromium
DQA data quality assessment
dynes/cm dynes/centimeter

LC/MS/MS liquid chromatography/mass spectrometry

LCS laboratory control samples

MACT Maximum Achievable Control Technology

MDH Minnesota Department of Health

MDL method detection limit

MPCA Minnesota Pollution Control Agency

MS matrix spike

MSD matrix spike duplicates

NEORSD Northeast Ohio Regional Sewer District

ng/l nanograms per liter

OAQPS U.S. EPA - Office of Air Quality Planning and Standards

OSHA Occupational Safety and Health Administration

PFBA perfluorobutanoate
PFBS perfluorobutane sulfonate
PFCs perfluorinated chemicals
PFDA perfluorodecanoate
PFDoA perfluorododecanoate
PFHpA perfluoroheptanoate
PFHxA perfluorohexanoate

PFHxS perfluorohexane sulfonate

PFOA perfluorooctanoate

PFOS perfluorooctane sulfonate PFOSA perfluorooctane sulfonamide

PFNA perfluorononanoate PFPeA perfluoropentanoate PFUnA perfluoroundecanoate

POTW publicly Owned Treatment Works

ppt parts per trillion

PQL practical quantification limit
QAPP Quality Asssurance Project Plan
R5 U.S. EPA - Region 5 office
RPD relative percent difference

U.S. EPA United States Environmental Protection Agency

WWTP wastewater treatment plant

# **EXECUTIVE SUMMARY**

In June 2008, R5 conducted a study to examine whether detectable levels of PFOS may be in the effluent of decorative chromium electroplating facilities that discharged to WWTPs. A year earlier, the State of Minnesota found high levels of PFOS at the Brainerd, Minnesota WWTP, and identified a chromium electroplating facility (Keystone Automotive) as the source [1]. Based on the State of Minnesota's findings, R5 initiated this study to investigate whether releases from chromium electroplating facilities could be a widespread source of PFOS in the environment. Along with other data, R5's study will be considered by the OAQPS to evaluate the use of PFOS in suppressing Cr(VI) emissions under air standards for this industry.

Samples were taken from seven Chicago, Illinois (Chicago), and four Cleveland, Ohio (Cleveland) facilities. R5 tested for thirteen PFCs, including PFOS, and data showed the following:

- PFCs were discharged from all eleven facilities' waste streams at quantifiable levels above background.
- "Background" was defined by the rinse water measurements. All eleven facilities used municipal tap water for their rinse water. Therefore, one rinse water (background) sample was taken in each city as a measurement of background PFC levels. The background PFOS level for Cleveland was 5.75 ppt. The background PFOS level for Chicago was 2.52 ppt.
- Ten out of the eleven facilities had PFOS detected in their wastewater in concentrations ranging from 31.4-39,000 ppt.
- Of the ten facilities with PFOS detections, none had effluent levels higher than those found at Keystone Automotive facility located near Brainerd, Minnesota.

### INTRODUCTION

In 2007, the MPCA tested the influent, effluent, and sludge at WWTPs across the State for PFCs, including PFOS. This investigation was done in order to determine if PFCs were present in these waste streams, and could therefore be a source of PFCs to the broader environment [1]. PFCs had been manufactured in Minnesota by 3M since the 1950s. 3M phased out the manufacturing of PFOS-related products in 2002 because of the growing research findings that PFOS was toxic to animals, persistent in humans, and widespread in the environment. In 2004, PFCs were detected in drinking water supplies in several eastern Twin Cities communities and traced to the legal disposal of 3M waste [2]. Through broader investigations, MPCA found widespread PFC contamination in various environmental media, including places with no known PFC sources.

Through testing at WWTPs, MPCA found relatively high levels of PFOS at the WWTP in Brainerd, Minnesota. The city of Brainerd is located about 135 miles northwest of St. Paul, along the Mississippi River. The initial 2007 sampling results at Brainerd were:

Influent: 811 ppt PFOS; Effluent: 1500 ppt PFOS;

Sludge: 861,000 ppt PFOS [1].

WWTP effluent may be a significant entry of PFCs to the environment [3], and several studies have concluded that conventional wastewater treatment may not be effective in removing these compounds [4] [5].

MPCA traced the PFOS in Brainerd's WWTP to a local chromium electroplating facility, Keystone Automotive (Keystone). Keystone was reportedly one of the largest chrome bumper repair and plating facilities in the United States. Since 1995, Keystone had been applying a commonly used PFOS-containing mist suppressant (Fumetrol 140®) in order to comply with the CAA's Cr(VI) MACT standard. As a result of MPCA's findings, the company switched to an alternate non-PFOS containing mist suppressant in early September 2007 [1].

#### Background

Cr(VI) electroplating is the electrical application of a coating of chromium onto a surface for decoration, corrosion protection, or durability. An electrical charge is applied to a tank (bath) containing an electrolytic salt solution. The electrical charge causes the chromium metal in the bath to fall out of solution and deposit onto objects placed into the plating bath. In an anodizing process, an oxide film is formed on the surface of the part. These electrolytic processes cause mist and bubbles containing Cr(VI) to be ejected from the bath, released into the work place, and eventually dispersed into outdoor ambient air unless controlled with add-on air pollution control equipment or chemical fume suppressants.

Chemical fume suppressants reduce surface tension and thereby, control Cr(VI) emissions. Surface tension is the force that keeps a fluid together at the air/fluid interface, and typically is expressed in force per unit of width, such as dynes/cm. By reducing surface tension in the plating/anodizing bath, gas bubbles become smaller, and rise more slowly than larger bubbles. Slower bubbles have reduced kinetic energy so that when the bubbles do burst at the surface, the Cr(VI) is less likely to be emitted into the air, and the droplets fall back onto the surface of the bath [6]. Ideally, chromium plating baths should have surface tension values between 45-55 dynes/cm [7].

Cr(VI) is a human carcinogen. Therefore, the U.S. EPA regulates Cr(VI) electroplating or Cr(VI) anodizing tank operations by applying the CAA MACT limits. The MACT limits require control of Cr(VI) emissions to the atmosphere by either limiting the amount of Cr(VI) through use of add-on air pollution control devices or utilizing a chemical fume suppressant [8]. These facilities are also regulated by OSHA under 29 CFR Part 1910.1026 to protect workers from occupational Cr(VI) exposure. Employers are required to use engineering and work practice controls to reduce and maintain employee exposure to Cr(VI) at or below the permissible exposure level of 5 micrograms per cubic meter of air, calculated as an 8 hour time weighted average [6] [8].

# Purpose of Study

After the release of MPCA's findings, R5 examined whether the release of PFOS through normal electroplating operations to WWTPs was a widespread or isolated event. Conversations between R5 staff and the Metal Refinishers Association indicated that PFOS use had become the industry standard as the most economic method of complying with the MACT rule [9]. Additionally, a 2003 survey conducted by the CARB, found that 190 of the 222 Cr(VI) electroplating operations in California used a fume suppressant, either in part or solely, to control Cr(VI) emissions. Almost all of the 190 operations used a chemical fume suppressant with PFOS as the active ingredient, and 124 reported using the same suppressant (Fumetrol 140®) that Keystone used. [6].

R5 provided this information to OAQPS. OAQPS was preparing to conduct a residual risk assessment for Cr(VI) electroplating, and to collect data through the ANPRM. Typically, releases of PFOS compounds would not be considered during a residual risk review since it is not one of the listed 188 hazardous air pollutants as defined by the Clean Air Act. However, OAQPS agreed to use the ANPRM to review data on the extent of PFOS mist suppressant use in Cr(VI) electroplating facilities and the potential release to WWTPs.

Because available data were likely to be limited, R5 also decided to gather data for OAQPS through a study to evaluate whether detectable levels of PFOS were present in the effluent of decorative Cr(VI) electroplating facilities that discharged to publicly owned WWTPs.

# PROJECT DESCRIPTION

During June 2008, R5 collected samples of discharged process wastewaters at eleven decorative Cr(VI) electroplating facilities. The study was confined to facilities in the Chicago and Cleveland areas. Only decorative, rather than hard, chromium electroplaters were selected because of their higher likelihood to generate wastewaters that would then be discharged to a WWTP [10].

#### Site Selection

Prior to sampling, field investigators conducted telephone surveys of chromium electroplating and chromic acid anodizing facilities to determine the best candidates. The Cleveland area list of potential study candidates was assembled by identifying facilities with chromium emissions reported in the Aerometric Information Retrieval System, and facilities that were subject to the Electroplating Point Source Category at 40 CFR 413. The latter group of facilities was provided by the NEORSD, which operates the three area POTWs, and is the control authority for indirect dischargers in the Cleveland area.

The combined list contained sixty-four potential study candidates. Twenty-two candidates were contacted by telephone and asked if they: (1) performed Cr(VI) electroplating or anodizing; (2) discharged process wastewater; and (3) used chemical fume suppressants. If a company met these criteria, it was given a brief description of the project and told that the Cleveland team may sample their wastewater discharge as part of the project. Seven of the twenty-two facilities screened by telephone met all three criteria, and due to funding limitations; only four facilities that were furthest along in arrangements were selected (see **Figure 1**).

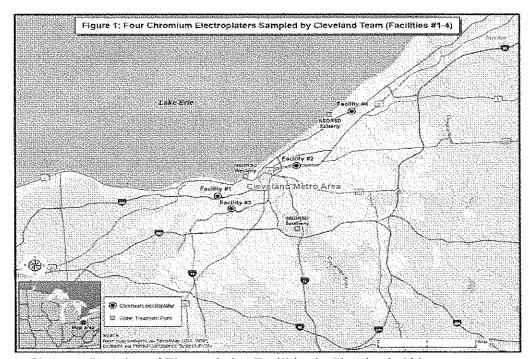


Figure 1: Location of Electroplating Facilities in Cleveland, Ohio

The Chicago area list of potential candidates was assembled by identifying facilities with existing air permits issued by the State of Illinois for operation of Cr(VI) electroplating tanks. The initial list contained twenty-six potential study candidates. Each of the twenty-six facilities were telephoned and asked the same questions as the Cleveland facilities. In addition, presampling site visits were conducted following the telephone surveys. Seven Chicago facilities were picked as final candidates (see **Figure 2**).

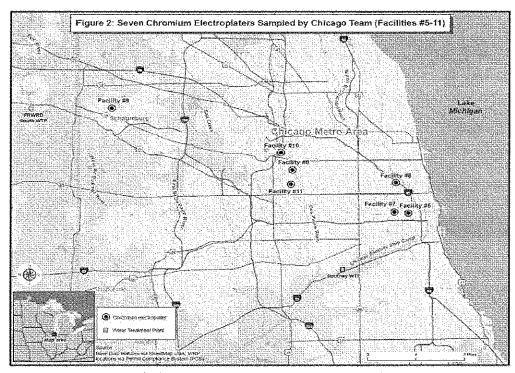


Figure 2: Location of Electroplaters Sampled in Chicago, Illinois.

All of the Chicago and Cleveland facilities selected performed decorative Cr(VI) electroplating on metal and/or plastic. Each facility used chemical fume suppressants (wetting agent and/or foam blanket) in its chromic acid bath tank to comply with the Cr(VI) electroplating MACT. In addition to chemical fume suppressants, two Cleveland facilities also employed add-on air pollution control devices. It was noted that facilities plating on plastic also used wetting agents in their chrome etch tanks for process control (i.e. to prevent voids in corners and creases of parts).

#### Facility Inspections

Both field investigation teams conducted cursory inspections at the facilities in conjunction with the sampling. During the inspections, the plating process, wastewater treatment, water usage, and usage of chemical fume suppressants were reviewed. Details of the inspections are summarized in **Appendix B-D**.

#### Sampling

The Cleveland field investigators collected samples between June 9 and 12, 2008, and the Chicago field investigator collected samples on June 9, 2008. All samples were taken during normal plating operations. At least one sample of discharged process wastewater from each facility was collected immediately prior to entry into the public sewerage system. Discharged process wastewater was comprised of the treated rinse waters from the plating operations, not including sanitary wastewater. In addition to the single effluent sample collected at each facility, the field investigators collected additional quality control samples at one select facility. These additional samples included the rinse water (background sample), field blank, and effluent duplicate. Samples were collected directly into laboratory-provided containers using standard operating procedures. The field blank was obtained by pouring reagent grade water into a laboratory-provided container while adjacent to the facility's discharge location. Samples were placed into iced coolers, refrigerated under custody until shipment to the laboratory, and cooled with blue ice packs during shipment. The samples were subsequently analyzed by AXYS Analytical Services Ltd. of Sidney, British Columbia, Canada, for thirteen PFCs, including PFOS (see Figure 3 for results). The analytical method used was solid phase extraction with High Performance Liquid Chromatography, tandem mass spectrometry (LC/MS/MS).

 $\underline{RESULTS}$ 

Figure 3: PFC Concentrations at Sampled Electroplater Facilities (reported in ppt)

Facility ID#: Fume Suppressant(s)	PFBA.	PFPeA	PFHxA	РЕНрА	PFOA	PFNA	PFDA	PFUnA	PFD <sub>0</sub> A	PFBS	PFHxS	PFOS	PFOSA	Sum of PFCs	PFOS/ PFC
Facility #1: Mist Suppressant A, B, C	9.06	42.6	90.7	56.2	83.3	ND	ND	ND	ND	9,160	67.8	31,100	ND	40,610	77%
Facility #2: Mist Suppressant D, E	48.3	30.9	·ND	ND	ND	ND	ND	ND	ND	41,800	306	708	ND	42,893	2%
Facility #3: Mist Suppressant B, F	ND	ND	177	175	650	13,100	27.1	44.1	ND	75.5	ND	ND	ND	14,249	<0.26%
Facility #4: Mist Suppressant D	ND	ND	ND	ND	ND	ND	ND	ND	ND	15,600	ND	39,000	ND	54,600	71%
Facility #5: Mist Suppressant G	ND	ND	ND	ND	ND	ND	ND	ND	ND	1,010	ND	2,320	ND	3,330	70%
Facility #6: Mist Suppressant Unknown	ND	ND	ND	ND	4.02	ND	ND	ND	ND	1,570	16.3	1,380	ND	2,970	46%
Facility #7: Mist Suppressant H	ND	1.08	ND	ND	3,11	ND	ND	ND	ND .	ND	ND	301	ND	305	99%
Facility #8: Mist Suppressant H	ND	ND	2.3	1.17	3.17	ND	ND	ND	ND	311	993	1,770	ND	3,081	57%
Facility #9: Mist Suppressant Unknown	ND	ND	ND	ND	1.73	ND	ND	ND	ND	2,250	163	4,460	ND	6,875	65%
Facility #10: Mist Suppressant Unknown	1.54	1.29	1.82	ND	3.32	ND	ND	ND	ND	ND	3.53	31.4	ND	42.9	73%
Facility #11: Mist Suppressant Unknown	14.3	ND	ND	ND	ND	ND	ND	ND	ND	1,510	9,430	1,260.	ND	12,214	10%
Number of Detects	4	4	4	3	7	1	1	1	0	9	7	10	0	11	
Minimum	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	31.4	ND	42.9	
Maximum	48.3	42.6	177	175	650	13,100	27.1	44.1	ND	41,800	9,430	39,000	ND	54,600	
Cleveland Background Sample	1.42	1.58	ND	1.74	2.19	ND	ND	ND	ND	ND	ND	5.75	ND		
Chicago Background Sample	ND	ND	ND	ND	1.37	ND	ND	ND	ND	ND	ND	2.52	ND		

 $\frac{Notes}{ND \ means that the analyte was not detected at the method detection limit.} \\ ND \ ranged \ from < 1.00 \ to < 45.3 \ ppt \ depending on analyte.}$ 

For total PFCs, a value of zero was used in the sum of PFCs calculation.

# **DISCUSSION**

Data results summarized in Figure 3 showed the following.

• PFCs were discharged from all eleven facilities' waste streams at quantifiable levels above background. Ten out of the eleven facilities had PFOS above background detected in their waste discharge streams (**Figure 4**).

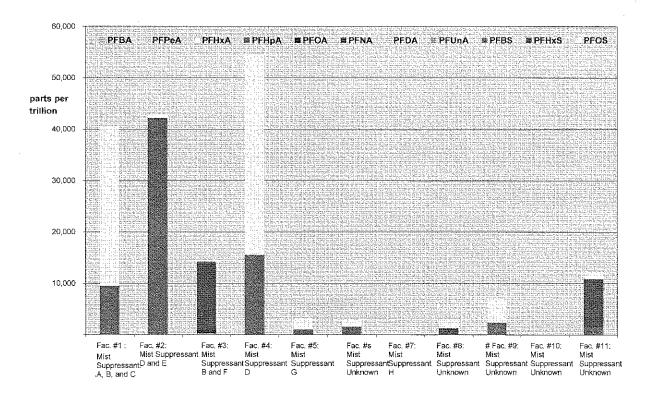


Figure 4: PFC Concentrations in Effluent of Electroplaters Using Chemical Fume Suppressants

• "Background" was defined by the rinse water measurements. All eleven facilities used municipal tap water for their rinse water. Therefore, one rinse water (background) sample was taken in each city as a measurement of background PFC levels. The background PFOS level for Cleveland was 5.75 ppt. The background PFOS level for Chicago was 2.52 ppt. In addition to PFOS, four other PFCs were detected in the Cleveland background sample, and one other PFC was detected in the Chicago background sample.

• At least eight different mist suppressants or mixture of suppressants were used at the various facilities and are as followed (several facilities did not provide information).

Benchmark Benchbrite STX AB (custom-made)
Benchmark Benchbrite STX
Benchmark CFS
MacDermid Proquel B
MacDermid Macuplex STR
Plating Process Systems PMS-R
Fumetrol-140
Brite Guard AF-1 fume control.

- Of the ten facilities with PFOS detections, none had effluent levels higher than those found at Keystone. In a sample dated December 2007, Keystone had a PFOS result of 278,000 ppt. [1]. The highest effluent PFOS result in this study was 39,000 ppt.
- The averages of the four highest concentrated compounds were: PFOS at 7680 ppt; PFBS at 6580 ppb; PFNA at 1190 ppt; and PFHxS at 1100 ppt (these averages were calculated using zero for the nondetects). These four chemicals made up over 99% of all compounds (Figure 5). PFOS, PFBS, PFHxS, and PFOA were the most commonly detected PFCs.

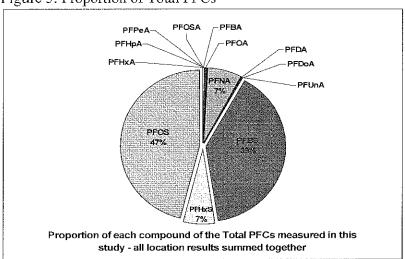


Figure 5: Proportion of Total PFCs

- Another PFC compound of general interest, PFOA, was detected at seven of the eleven facilities, ranging from 1.73 650 ppt.
- PFCs were found in one field blank and in the background samples. Field blanks
  consisted of reagent grade bottled water exposed to the atmosphere at the designated
  facility. The field blank with PFHxS detection was exposed at facility #11 whose
  effluent samples contained PFHxS concentrations at the highest levels detected in this
  study. We attributed PFCs in the background samples to trace background levels found

in municipal tap water. Lake Michigan is the source water for the Chicago Municipal Utilities, and Lake Erie is the source water for the Cleveland Municipal Utilities. During the time of this study, both utilities were in compliance with all federal and state drinking water standards.

• Although not applicable to this industry, we compared our results to state and federal PFC guidance levels. Nine of the ten facilities tested above the U.S. EPA provisional health advisory for PFOS in drinking water set at 200 ppt. Ten of the eleven facilities tested above the Minnesota water quality criteria for PFOS in the Mississippi River (6 ppt).

# Conclusions

The data clearly indicated that decorative chromium electroplaters discharge PFOS and other PFCs to WWTPs in concentrations higher than background levels. Data also indicated that mist suppressants have very specific PFC mixtures, which may be found in the resulting electroplater effluent. The concentrations vary widely which is most likely due to the inherit design of study. Therefore, care should be taken when comparing results from one facility to another, as the study included facilities of different operational sizes and production schedules. Facilities also varied widely in the brand of mist suppressant used, and amount added to the plating baths.

We would like to emphasize the nexus between the PFOS emissions and the Chromium MACT rule. To comply with the MACT rule, many facilities have chosen PFOS-containing mist suppressants as the best available technology to achieve Cr(VI) risk reduction in lieu of adding control technology. EPA believes that the PFOS emissions (as well as other PFC emissions reported in this survey) should provide target areas for improved pollution prevention performance including: (1) the development of alternative PFC free mist suppressants; (2) the improved procedures to reduce and capture downstream PFC levels in the wastewater prior to release into the waste water treatment facility; and (3) enhancing operating processes that limit the amount of PFC added to plating baths to efficaciously promote plating while reducing PFC total consumption.

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# **APPENDIXES**

# Appendix A - Data Quality Assessment Report

#### INTRODUCTION

This report was developed as a scientific evaluation designed to determine if the PFOS data obtained from the 2008 R5 Electroplater PFOS study were appropriate to meet the study objectives, and were of the right type, quality, and quantity to support the intended use. This assessment also estimated the level of confidence attributable to the data set. In brief, our analyses showed that some decisions and conclusions associated with these data could be made with a high degree of confidence, while other decisions had significant limitations associated with them.

The data were used to evaluate wastewater contamination associated with average industrial chromium decorative electroplaters. This PFOS study did not include a statistical sample design, and as such, rigorous statistical evaluations were not used. The data were assessed using the following criteria.

- 1. Review the data quality objectives
- 2. Conduct a preliminary data review
- 3. Perform an analysis of the data
- 4. Verify the assumption of the analysis
- 5. Draw conclusion from the data

### 1. REVIEW OF DATA QUALITY OBJECTIVES

**Statement of the problem:** The overall objective of this study was to evaluate potential wastewater PFOS release associated with decorative chromium electroplaters.

#### Study question:

- 1) Was there PFOS in the wastewater discharge to WWTPs from decorative chromium electroplaters?
- 2) Were these discharges quantifiable?

### Identification of the decision:

Decision statement -

If PFOS discharges from decorative chrome electroplaters are present at facilities using an approved MACT standard technology for suppressing Cr(VI) emissions, then these results may be useful in informing the OAQPS rulemaking process.

#### Identification of inputs to the decision:

Facilities were selected based on:

- Use of PFOS containing surfactant; and
- Potential for rinse stream/waste water contamination.

#### Definitions of the boundaries for the study:

This study was confined to chromium electroplating facilities in the Chicago and Cleveland areas. Samples were taken during normal plant operating conditions, and sampling locations were representative of discharged wastewater to POTWs.

#### Documented decision rules:

- PFOS was present when the analyzed concentration was above the laboratory MDL.
- PFOS discharges were quantifiable when effluent concentrations observed were above the laboratory PQL.
- PFOS discharges were believed attributable to the use of MACT complaint Cr(VI) suppressants, when effluent concentrations were above background PFOS levels.

# Optimize the design for obtaining data:

This analysis may be useful in addressing future studies of PFOS and other PFCs as related to wastewater discharges.

#### 2 PRELIMINARY DATA REVIEW

#### Completeness

All samples identified in the QAPP were collected and analyzed.

#### **Holding Time**

All samples were analyzed within the required holding times.

### **Sample Preservation**

All samples were collected and iced for shipment to lab.

# Sample Receipt

All samples were received on ice within 24 hours of shipping. The samples were all refrigerated at 4 degree C prior to extraction and analysis.

#### Sample Extraction and Analysis

Samples were analyzed in three batches. Sample extraction, instrumental analysis, and analyte quantification procedures were in accordance with the lab's standard operating procedures. Samples were spiked with 13C-labelled quantification standards and extracted and cleaned up using SPE cartridges. Extracts were instrumentally analyzed using liquid chromatography/mass spectrometry (LC/MS/MS). Analyte concentrations were determined by isotope dilution/internal standard quantification. Reporting limits were defined as the concentration equivalent to the lowest calibration standard or the sample specific detection limit, whichever was greater.

#### **Instrument Calibration**

All initial calibrations specifications were reported as met. Initial calibration percent recoveries and retention times demonstrate ongoing precision and accuracy.

#### **Continuing Calibration**

All continuing calibration and verification specification were met.

#### Internal standards

All ongoing precision and recovery specifications were met.

# **Target Compound Results**

No apparent matrix interferences were noted in the analysis of the target compounds. Sample analyte concentrations were not blank corrected and results should be evaluated with consideration of the procedural blank results.

# 3. Data Analysis

In this study, the MDL was used to determine if an analyte was present in a sample and the PQL was used to make a quantitative determination of the amount of analyte in the sample. The U.S. EPA uses the term MDL and PQL to describe the specific approaches of estimating the detection and quantification limits, respectively. If comparing concentration directly to a standard, it must be greater than the quantification limit in order to provide a reliable estimate whether or not the standard has actually been exceeded. To determine whether or not an analyte is present or absent in a sample, a result will be above the detection limit. Measurements above the quantification limit can be used directly. Measurements below the quantification limit are considered censored and must be appropriately adjusted.

The blank data from this experiment showed rather conclusively that PFOS concentrations found in the electroplater effluents were tied to PFOS-containing suppressants. It is also unlikely that PFOS or other PFCs were introduced at significant levels through other means. Laboratory blank samples were free of contamination. Field blanks, consisting of commercially available reagent water, were also free of significant contamination. Facility source waters were analyzed to evaluate potential contamination coming into the facilities. These samples were also free of significant PFOS concentrations. Surrogate recoveries for the lab, field, and source water blanks ranged from 69 to 148% and averaged 105%, demonstrating that sample preparation and analyses were free of contamination.

Analysis of Quality Control Background PFOS Sampling (in ppt)

Blanks	Analyses	Min	Max
Laboratory	3	0	0
Field	2	0	0
Source Water	2	2.52	5.75

Note: Lab blanks were free of contamination.

One field blank contained trace level of PFHxS.

Source water samples contained trace levels of PFOS and other PFCs.

#### Laboratory Control Samples

Three laboratory control samples were analyzed for the Chicago and Cleveland batch of samples as well as the re-analysis of selected samples. PFOS recoveries ranged from 89 to 105%, and had an average recovery of 96% (see below).

Sample	A-Ohio	B-Illinois	C-Illinois		
Name					
Analyte	% Recovery	% Recovery	% Recovery	AVG	%RPD
PFBA	77.3	107	85.3	90	33
PFPeA	87.1	109	94	97	23
PFHxA	84	119	101	101	35
PFHpA	76.8	117	102	99	. 41
PFOA	84.4	112	78.1	92	37
PFNA	89.9	96.6	99.2	95	10
PFDA	104	98.8	118	107	18
PFUnA	107	94.2	118	106	22
PFDoA	86.1	119	95.5	100	33
PFBS	102	110	113	108	10
PFHxS	85	119	101	102	33
PFOS	89.2	105	94.2	96	16
PFOSA	94.7	100	109	101	14

# Matrix Spike Duplicates

Matrix spike and spike duplicate analyses were performed to evaluate the potential for sample interferences. Matrix interferences are also referred to as matrix effects. Matrix spike interferences are those chemical and/or physical interferences that impede the analytical instrumentation in detecting the true value concentration of a target analyte within a sample. One possible source of matrix interferences may be caused by contaminants that are co-extracted from the sample and result in a positive or negative bias. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the sample matrix.

	MATRIX SPIKE	MATRIX SPIKE DUPLICATE	3	
	Illinois	Illinois	·	
ID .	Matrix Spike I	Matrix Spike Duplicate I		
Sample Size	0.0349 L	0.0318 L		
Analyte	% Recovery	% Recovery	AVG	%RPD
PFBA	101	89.6	95	12
PFPeA	92.4	120	106	26
PFHxA	101	114	108	12
PFHpA	99	109	104	10
PFOA	81.8	122	102	39
PFNA	116	112	114	4
PFDA	99.1	90.1	95	9
PFUnA	76.9	88.7	83	14
PFDoA	93.5	87	90	7 ·
PFBS	104	109	107	5
PFHxS	98.2	101	100	3
PFOS	95.8	105	100	9
PFOSA	105	114	110	8

Spike recoveries for PFOS ranged from 96 to 105 averaging 100% with a 9% RPD. While these recoveries were within laboratory specifications, the spiking concentrations were well above the sample concentration. This practice did not allow an appropriate assessment of the impacts from the sample [see comparison table of laboratory control samples (LCS) and matrix spike(MS) /matrix spike duplicates (MSD) %RPDs below].

Comparison of Precision between LCS and MS/MSD

	MS/MSD	LCS
Analyte	%RPD	%RPD
PFBA	12	33
PFPeA	26	23
PFHxA	12	35
PFHpA	10	41
PFOA	39	37
PFNA	4	10
PFDA	9	18
PFUnA	14	22
PFDoA	7	33
PFBS	5	10
PFHxS	3	33
PFOS	9	16
PFOSA	8	14

#### Field Duplicates

A field duplicate is a duplicate sample collected by the same team or by another sampler or team at the same place, at the same time. It is used to estimate sampling and laboratory analysis precision. PFOS duplicate analyses ranged from 40 to 96% RPD and demonstrated variable

precision for the selected samples. Values for RPD appeared dependent on the concentrations found in the sample. Analysis of sample with low concentration of PFOS appeared more precise (i.e., lower RPD). Samples with higher concentration of PFOS appear less precise (i.e., higher RPD). The laboratory narrative report from AYXS attributes these variable recoveries to the presence of particulate matter in the subject samples. Given the limited number of samples collected and the general expectation that PFOS will attach to particulate matter, future analyses should ensure greater homogenization of samples or collection of sufficient samples such that statistical evaluations may be conducted. Based on the particulate-free LCS results and their acceptable precision and accuracy, we believe the variability in wastewater sample recoveries do not impinge our study conclusions.

Field Duplicate Results

		Cleveland	&	Chicago	Dup	Sampling	
ID		Cievelanu	· ·	Cincago	Dup	Jampinig	
Sample size	0.0163 L	0.0154 L		0.0658 L	0.0727 L	0.0718 L	
Analytes	ppt	ppt	%RPD	ppŧ	ppt	ppt	%RPD
PFBA	48.3	45.6	6	< 7.60	8.6	17.1	66
PFPeA	30.9	33.4	8	8.29	9.93	7.4	30
PFHxA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFHpA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFOA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFNA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFDA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFUnA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFDoA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0
PFBS	41800	39900	5	1410	1580	1820	26
PFHxS	306	227	30	8900	11400	12600	34
PFOS	708	470	40	2040	6180	4680	96
PFOSA	< 30.6	< 32.4	0	< 7.60	< 6.88	< 6.97	0

#### Surrogate Spikes

A surrogate is a pure analyte that is extremely unlikely to be found in any sample. It is added to a sample aliquot in known amounts before extraction and is measured with the same procedure used to measure other sample components. A surrogate behaves similarly to the target analyte and most often used with organic analytical procedures. The purpose of a surrogate analyte is to monitor method performance with each sample. This study used 7- C 13 substituted isotopes.

For all samples collected, surrogates recoveries ranged from 25 to 148% and averaged 91%. These recoveries were within historical laboratory specifications and analyses were generally within control. For the analyte specific surrogate, 13-C4 PFOS recoveries ranged from 53.1 to 132% and averaged 90%. These recoveries demonstrate acceptable precision and accuracies for evaluating the target compound.

		Surrogate	Percent	Recoveries							
Sample ID	A	В	С	D	E	F	G	H	I	J	K
LABELED COMPOUND											
13C4-PFBA	115	119	118	79.6	24.7	28	84.6	47	78.2	46.9	105
13C2-PFHxA	107	124	114	99.2	94.2	40	95.7	80.5	91.4	61.5	115
13C2-PFOA	138	113	116	101	109	106	85.9	109	113	93.4	104
13C5-PFNA	77.8	147	66	137	126	66.4	96.6	78.2	85.7	69.9	111
13C2-PFDA	75.4	124	108	69.5	90.3	43.7	84.9	80.9	66	82.2	78.8
13C2-PFDoA	111	148	83.7	84.7	114	69.7	60.2	89.2	74.5	73.4	86.8
13C4-PFOS (80)	100	132	132	83	82.7	53.1	93.8	70.6	55.7	72.6	103

# **PFOS/PFC Suppressant Analysis**

Eleven facilities were sampled and analyzed for the presence of PFOS, and other PFCs. A review of facility records showed that at least eight different suppressants or mixtures were used at the various facilities. Several facilities did not provide information on the suppressant used. Ten facilities had PFOS wastewater sample results above the MDL. For this study, the PQL was defined as 5 times the MDL. The positive PFOS results ranged from 231 to 2976 % of the calculated sample PQLs.

Facility Results Compared To Quantitative Definitions

Facility	AVG Sample size	PFOS (ppt)	Sample specific MDL	PQL (5XMDL)	%>PQL
#1	0.0619 L	31100	209	1045	2977
#2	0.0163 L	708	61.2	306	231
#3	0.0269 L	U	37.1	185	0
#4	0.0601 L	39000	423	2115	1844
#5	0.200 L	2320	42.9	214.5	1082
#6	0.201 L	1380	11.8	59	2339
#7	0.498 L	301	9.13	45.7	659
#8	0.494 L	1770	32.1	161	1099
#9	0.350 L	4460	97.5	488	914
#10	.497 L	31.4	2.01	10.05	312
#11	.0718 L	4680	33	165	2836

#### 4. VERIFICATION OF ASSUMPTIONS

We have verified the following assumption in evaluating our study question, "Is PFOS discharged from decorative chrome plating operations?"

- Various Cr(VI) control methods are available;
- PFC-containing mist suppressants are in common industry use;

- Composition of suppressants may include various PFOS formulations;
- Active suppressant ingredients contain other PFCs beyond PFOS;
- Suppressant application is monitored;
- Electroplating discharges are amenable to PFOS analysis; and
- PFOS analyses are reasonably precise, accurate, recoverable, and reproducible.

#### 5. SUMMARY OF CONCLUSIONS

Several conclusions can be made from the data collected:

- ✓ The small sample size limits the ability to draw conclusions beyond the observation that PFOS as well as other PFCs appear to be discharged from decorative chromium electroplating facilities through wastewater discharge;
- ✓ These discharges are quantifiable;
- ✓ Composition of PFOS containing mist suppressant vary widely;
- ✓ Variability in wastewater sample recoveries do not impinge our study conclusions; and
- ✓ PFOS data obtained from this study were appropriate to meet the study objective, and were of the right type, quality, and quantity to support the intended use.

Appendix B. Facility Operations

	Facility #1	Facility #2	Facility # 3	Facility # 4
Plating and metal finishing operations	Two lines of decorative chrome electroplating with chromic acid on plastic parts. A third plating line applies a gold-colored finish to nickel-plated parts.	Decorative chrome electroplating with chromic acid on metal and plastic parts. One line uses plastic parts and one uses metal parts.	Decorative chrome electroplating with chromic acid on plastic parts.	Decorative chrome electroplating with chromic acid on metal parts (brass, steel, and stainless steel), cadmium plating, and chromate conversion coating.
Operating schedule	24 hours/day, 5 days/week	Plastic substrate parts line: 24 hours/day, 5 days/week  Metal substrate parts line: 8 hours/day, 5 days/week	24 hours/day, 5 days/week	10 hours/day, 4 days/week
Chromium electroplating process description	Parts are prepared by dipping in a chromic acid etch bath, neutralization tank, palladium-tin activator bath, and accelerator to remove tin. The plating process includes copper strike, bright acid copper, electroless nickel (and semi-bright, high sulfur and bright nickel for exterior use parts), and chromium electroplating with chromic acid. Each step is followed with rinsing.	Plastic substrate parts line: Parts are prepared by dipping in a chromic acid etch bath, neutralization tank, activator, and accelerator. The plating process includes electroless copper, copper strike, bright acid copper, nickel, and chromium electroplating with chromic acid. Each step is followed by rinsing.  Metal substrate parts line: Parts are prepared by dipping in a cleaner and then an acid tank. The plating process includes copper strike, bright acid copper, nickel, and chromium electroplating with chromic acid. Each step is followed with rinsing.	Parts are prepared by dipping in a chromic acid etch bath, neutralization tank, activator, and accelerator. The plating process includes electroless copper, copper strike, bright acid copper, semibright or satin nickel, bright nickel, microporous nickel, and chromium electroplating with chromic acid. Each step is followed with rinsing.	Parts are prepared by dipping in a soak cleaner, electrocleaner, and then sulfuric acid (with or without current). The plating process includes nickel strike, bright nickel, and chromium electroplating with chromic acid. Each step is followed with rinsing.

	Facility #5	Facility #6	Facility #7	Facility #8	Facility #9	Facility #10	Facility #11
Plating and metal finishing operations	This facility operates two chrome tank lines which are 225-gallons each.		This facility operates a single 350-gallon chrome tank.	This facility operates a single 400- gallon chrome tank.	This facility operates a 500-gallon chrome tank.	This company operates a 500-gallon chrome tank.	This facility operates a large 4000-gallon chrome tank. The facility decorates a variety of metal parts including shopping carts and other pieces.
Operating schedule		This facility has not used its chrome tank for over sixty days and rarely chromes metal pieces.	2 hour/day, maximum of 15 hours/week.		1 day/week.	2-4 hours/day.	8 hours/day, 5 days/week.
Chromium electroplating process description							

Appendix C. Rinsing Practices, Pretreatment, and Wastewater Discharge

	Facility #1	Facility #2	Facility #3	Facility #4
Rinsing practice for chromium electroplated parts	The interior parts line has four city water rinses followed by one deionized water rinse. The exterior parts line has three city water rinses followed by one deionized water rinse. Rinsewaters flow counter-currently.	The metal substrate parts line has three city water rinses. Rinsewaters flow countercurrently. The plastic substrate parts line has four city water rinses followed by one dejonized water rinse. Rinsewaters flow countercurrently.	Two city water rinses followed by three deionized water rinses. Rinsewaters flow countercurrently.	One city water static rinse, three countercurrent city water rinses, then one deionized water static rinse. The final deionized water static rinse is emptied daily.
Rinse water pretreatment	Acid, chromium electroplating, electroless nickel, and copper-nickel rinsewaters are received in separate tanks in the pretreatment plant. Nickel is recovered by ion exchange before nickel rinsewaters are pumped to the pretreatment facilities. At the pretreatment plant, acid rinsewaters are combined with chrome-bearing rinsewaters. Chrome is reduced with sodium metabisulfite. Dissolved metals in electroless nickel rinsewaters are precipitated with calcium chloride. Chemically treated chromium and nickel wastewaters are pumped to the copper-nickel tank, where pH is adjusted with acid or caustic. Solids are settled in three clarifiers in series with the addition of a flocculent. Clarified wastewater is pumped to a storage tank with level control. When the tank level reaches a set point, wastewater is pumped to a sand filter. Filtered wastewater flows to a discharge tank. Clarifier sludge is pressed. Filtrate is returned to the copper-nickel tank.	Chrome-bearing rinsewaters are reduced by lowering pH with sulfuric acid and adding sodium metabisulfite. Reduced chrome rinsewaters are combined with other metal-bearing rinsewaters in the acid/alkali tank and neutralized with caustic. Solids are settled in a clarifier with the addition of flocculent polymer. Clarified wastewater flows through a surge tank and equalization tank. Wastewater then flows through a sand filter prior to discharge. Clarifier sludge is pressed. Filtrate is returned to the acid/alkali tank.	Chrome-bearing rinsewaters are reduced in a 4-stage tank by lowering pH to 2.5 s.u. with sulfuric acid and adding sodium metabisulfite. Other rinsewaters and calcium chloride are mixed with reduced chrome rinsewaters to raise the pH. Solids are settled in a clarifier with the addition of polymer. Clarified wastewater discharges from a flow-through final effluent tank. Clarifier sludge is thickened and pressed. Filtrate is returned to the clarifier. Electroless copper rinse water is treated separately by plating on steel wool.	Chrome-bearing rinsewaters (from chrome electroplating and chromate conversion) are reduced by lowering pH with sulfuric acid and adding sodium metabisulfite. Other rinsewaters are combined with reduced chrome rinsewaters and the pH is raised with sodium hydroxide. Solids are settled in a clarifier with the addition of polymer. Clarified wastewater is discharged. Clarifier sludge is dewatered in a filter press. Filtrate is returned to the clarifier. Cyanide plating rinse water is batch-treated with sodium hypochlorite, then combined with other rinsewaters.
Average wastewater discharge for operating days during June 2008	97,000 gal/day	29,400 gal/day	146,000 gal/day	6,700 gallons/day

<sup>&</sup>lt;sup>1</sup> Discharge flow data were provided by the supervisor of enforcement, water quality & industrial surveillance, Northeast Ohio Regional Sewer District. The companies' permits require that self-monitoring data be reported to NEORSD.

	Facility #1	Facility #2	Facility #3	Facility #4
POTW that receives wastewater	Northeast Ohio Regional Sewer District Westerly WWTP	Northeast Ohio Regional Sewer District Easterly WWTP	Northeast Ohio Regional Sewer District Easterly WWTP	Northeast Ohio Regional Sewer District Southerly WWTP
from facility POTW design	35 million gal/day	155 million gal/day	155 million gal/day	175 million gal/day
flow		133 mmon ganday	155 mmon gawaay	175 minon garday

	Facility #5	Facility #6	Facility #7	Facility #8	Facility #9	Facility #10	Facility #11
Rinsing	The chrome	Discharge flow	Rinse waters	After metal pieces	After metal	Rinse waters flow	They operate three rinse
practice for	pieces are dipped	through chrome	at the chrome	are chromed they	pieces are	are 5 gpm.	tanks (one counterflow).
chromium	into a single rinse	rinse tanks are	tank flow	pass thru two dead	chromed they		The make up rinse rate is
electroplated	tank. Rinse tank	1-2 gpm.	between 2-4	rinse tanks,	are dipped into		approximately 2 gpm.
parts	flows are		gpm.	followed by a	two static rinse	'	
	approximately 1-			running tank at 1-2	tanks,		
	2 gpm.			gpm.	followed by		
					three flowing		
					rinse tanks 0.5		
					gpm.		
Rinse water	Pretreatment	The facility's	All rinse	This facility	This facility	The facility operates	The electroplating shop
pretreatment	consists of a	pretreatment	waters flow	operates a	operates a	a complete	operates a complete
	series of	system consists	toa	complete	complete	pretreatment system	pretreatment system. It includes a chrome
	oxidations tank,	of an oxidation	pretreatment	pretreatment	pretreatment	consisting of chrome reduction	reduction tank, an
	followed by a	tank,	system. Entire flow	system consisting	system consisting of a	tank, flocculation	equalization tank, a pH
	flocculation tank,	flocculation	thru the	of a pH adjust with flocculation agent,	pH adjust with	tank, noccuration tank, clarifier, sand	adjust tank, a clarifier, and
	followed by a	tank, clarifier holding tank,		clarifier, final	flocculation	filters and pH adjust	finally, an effluent
	clarifier, then	then finally to	pretreatment system is 50	filtration then	agent, clarifier,	tank. Flow thru the	discharge pipe. Typical
	discharged to a sewer.	discharge.	-60 gpm.	discharge. Flows	final filtration,	pretreatment system	flows thru the pretreatment
	Sewei.	Flow thru entire	-oo gpiii.	thru the	then discharge.	is about 80 gpm.	system varies between 50 -
		pretreatment		pretreatment	Flow thru the	is about to Spin.	55 gpm
		system is		system are	pretreatment		33 8hm
		approximately		typically 50 -55	system is		
		20 gpm.		gpm.	about 55-55		
		20 gpm.		5P''''	gpm.	:	
Average							
wastewater							
discharge							
POTW that							
receives							
wastewater							
from facility							
POTW							
design flow							
	<u> </u>				[		

# Appendix D. Hexavalent Chromium Controls

	Facility #1	Facility #2	Facility #3	Facility #4
Chemical fume suppressant and add-on air pollution control devices used at facility	Mist Suppressant A, B and C	Mist Suppressant D and E	Mist Suppressant B and F	Mist Suppressant D
Tensiometer readings	Surface tension not greater than 35 dynes/cm as measured by a tensiometer	Surface tension not greater than 45 dynes/cm as measured by a stalagmometer	Surface tension not greater than 35 dynes/cm as measured by a tensiometer	Surface tension not greater than 45 dynes/cm as measured by a stalagmometer
Amount of chemical fume suppressant used	~2.6 gal/week of Mist Suppressant A, ~1.5 gal/week of suppressant B, and ~0.9 gal/week of suppressant C. <sup>2</sup>	1.2 gal/week of mist suppressant D and 3.5 gal/week of mist suppressant E. (Mist suppressant E usage is about 1.1 gal/week for the chromic acid tanks and 2.4 gal/week for the chrome etch tank.) <sup>3</sup>	8-9 gal/week of mist suppressant F. The usage of mist suppressant B was not determined.	0.06 gal/week of mist suppressant D.5

<sup>2</sup> The company will cease using mist suppressant C once it depletes its inventory.

These are average values based on the following usage -16 gallons, 15 gallons, and 15 gallons of mist suppressant D during the third and fourth quarters of 2007 and the first quarter of 2008; 47 gallons, 44 gallons, and 46 gallons of mist suppressant E during the third and fourth quarters of 2007 and the first quarter of 2008. 300 mL/day of mist suppressant E (0.4 gallons/week) are added to the chromic acid tank of the metal substrate line and 500 mL/day of Mist Suppressant E (0.7 gallons/week) are added to the chromic acid tank of the plastic substrate line based on surface tension logs. The remainder of suppressant E's usage is for the plastic substrate line chromic acid etch tank.

<sup>&</sup>lt;sup>4</sup> This value was given verbally by Facility #3 Director of Engineering during the inspection.

<sup>&</sup>lt;sup>5</sup> This is an average value calculated by summing the amount of chemical fume suppressant recorded on the company's surface tension log from February 19, 2007 through June 12, 2008 and dividing by the number of calendar weeks during that period.

	Facility #5	Facility #6	Facility #7	Facility #8	Facility #9	Facility #10	Facility #11
Chemical fume suppressant and add-on air pollution control devices used at facility	Mist Suppressant G	Unknown	Mist Suppressant H	Mist Suppressant H	Unknown	Unknown .	Unknown
Tensiometer readings	Recent tensiometer value of 44 dynes/cm2 was measured on June 7, 2008.	Latest tensiometer reading at chrome tank was measures at 43dynes/cm2 on April 10, 2008.	Latest tensiometer values of 23.6 dynes/cm2 were measured on June 2, 2008.	Latest tensiometer reading of 41dynes/cm2 was measured on June 9, 2008.	Last tensiometer reading was 27.3 dynes/cm2 as measured on June 3, 2008.	The last tensiometer value recorded at the chrome tank was 33.96 dynes/cm2 on May 29, 2008.	
Amount of chemical fume suppressant used							

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# Per- and Polyfluoroalkyl Substances (PFASs) Usage

NOTE: This document presents a compilation of information regarding known Per- and Polyfluoroalkyl Substances (PFASs) use and sources based upon a search of existing literature from a variety of sources (e.g., scientific journals, scientific publications, etc). EPA has compiled the information to help its regional offices and others identify possible contamination sources at sites with known or suspected PFAS releases. It may also help identify types of sites that may warrant further investigation for possible PFAS contamination. The compilation is entirely for informational purposes and is intended to serve as a general resource; readers should not construe it to be an exhaustive, definitive list. Decisions for investigation of PFAS releases at a site should be based on a site-specific determination in light of site-specific information. EPA anticipates updating the information as the Agency becomes aware of additional existing information or as new information becomes available.

PFASs are a class of man-made chemicals. They generally consist of a carbon backbone with fluorines saturating most of the carbons and at least one functional group, such as a carboxylic acid, sulfonate, amine, and others. If all the carbons except for the ones binding the functional group are saturated with fluorine, then the substance is called a perfluoroalkyl substance. If most but not all carbons are saturated with fluorine, then the substance is called a polyfluoroalkyl substance. Perfluorooctanoic acid (PFOA) and perfluorooctanesulfonate (PFOS) are the two most commonly known PFASs.

The list of uses below is not meant to indicate information about concentrations of PFAS within any applications. In some applications, the PFAS concentration within the product may have been de minimis, or an extremely tiny percentage. Unless stated, the information also does not indicate which specific PFAS was used or whether the usage was only in the past or is still currently being used.

#### • Fire-fighting foam

- o Numerous Superfund, federal facilities, and state-managed sites are known to have environmental media with PFAS contamination from this source.
- Aqueous film-forming foams (AFFF) are used to extinguish hydrocarbon fires at airports, train yards, oil refineries, and other locations. (UNEP 2011)
- Perfluorinated carboxylic acids manufactured by electrochemical fluorination were used as components in AFFF from about 1965-1975. Perfluorooctylsulfonyl fluoride (POSF CASRN 307-35-7)-based AFFF became the product of choice from the 1970s forward. (Prevedouros et al. 2006)
- PFASs are also used in dry fire-extinguishing agents to make powder nonwettable by hydrocarbons. (Kissa 2001)
- Metal plating and finishing

- Numerous sites are known to have environmental media with PFAS
  contamination from this source. In at least a few cases, the contamination appears
  to have come from air emissions.
- PFASs are used as a surfactant, wetting agent, and mist suppressing agent for chrome plating. It was previously used for decorative chrome plating, but new technology is making that obsolete. It may still be used in hard chrome plating. (UNEP 2011)
- PFAS use for second generation wetting agent fume suppresant (WA/FS) was first reported in the chromium plating industry in 1954. The original second generation WA/FS was a PFAS with an amino group. Later PFAS included potassium perfluoroalkyl sulfonate, amine perfluoroalkyl sulfonate, potassium perfluoroethyl cyclohexyl sulfonate (CASRN 67584-42-3), and ammonium perfluorohexylethylsulfonate (FtS 6:2, CASRN 59587-39-2). The third generation WA/FS which were introduced in the late 1980s/early 1990s also contain PFAS including organic fluorosulfonate and tetraethylammonium perfluoroctyl sulfonate (CASRN 56773-42-3). (EPA 1998)
- PFASs improve the quality of electroless plating of copper and stabilize coating baths for depositing nickel-boron layers. (Kissa 2001)
- o PFASs are used in electroplating of copper, nickel, and tin. It improves the stability of the baths and improves overall performance. PFASs can be used as a leveling agent for zinc electrodeposition. (Kissa 2001)
- PFASs can be used to treat metal surfaces to prevent corrosion, reduce mechanical wear, or enhance aesthetic appearance. They promote the flow of metal coatings and prevent cracks during drying. (Kissa 2001)
- o PFAS dispersion products, which are used to coat metals, have been manufactured since 1951. (Prevedouros et al. 2006)
- Some PFASs are effective blocking agents for aluminum foil. Monfluor 91 is a noted brand name for this. (Kissa 2001)
- Brand names associated with PFAS dust suppression include Fluorotenside-248, SurTec 960, and Fumetrol (ATOTECH).

#### Landfills

- Numerous sites are known to have environmental media with PFAS contamination from this source.
- o Landfills can be a source of PFASs if PFASs were deposited in the landfill.
- Landfills can also be a source of PFASs if certain PFAS polymers were placed in the landfill because some polymers can degrade to the monomers in landfills.
   (Washington and Jenkins 2015; Washington et al. 2015)

#### Textiles

PFASs are used extensively by the textile industry for their ability to repel oil, water, and stains. Many types of outwear such as jackets, shoes, and umbrellas are treated with PFASs. Household products such as carpets, upholstery, and leather are also treated. Outdoor equipment such as tents and sails are treated. (UNEP 2011)

- PFAS dispersion products, which are used to coat fabrics, have been manufactured since 1951. (Prevedouros et al. 2006)
- PFAS brand names associated with textiles include Scotchgard (3M) and Zonyl,
   Foraperle, and Capstone (DuPont)
- Paper and cardboard packaging
  - o PFASs are used to produce waterproof and greaseproof paper. Packaging includes food contact paper such as plates, popcorn bags, pizza boxes, and food containers and wraps. Packaging also includes non-food contact applications such as folding cartons, carbonless forms, and masking papers. (UNEP 2011) (Kissa 2001)
  - o PFAS brand names associated with packaging include Scotchban (3M), Baysize S (Bayer), Lodyne (Ciba, BASF), Cartafluor (Clariant), and Zonyl (DuPont).
- Industrial and household cleaning products
  - Because of their surfactant properties, PFASs have been used to lower surface tension and improve wetting and rinse-off in many industrial and household cleaning products such as carpet spot cleaners, alkaline cleaners, denture cleaners and shampoos, floor polish, and dishwashing liquids. They were also used in car wash products and automobile waxes. (UNEP 2011) PFAS in windshield wiper fluid prevents icing of the windshield. (Kissa 2001)
  - The can be used in cleaners containing strong acids and bases, including those for cleaning concrete, masonry, and metal surfaces (such as airplanes). PFAS in nonaqueous cleaning agents aid removal of adhesives and in dry cleaning of textiles or metal surfaces. Machine parts are cleaned after nickel plating with a solution containing PFOS. (Kissa 2001)
  - o PFASs are used in cleaning formulations that remove calcium sulfate scale from reverse osmosis membranes. (Kissa 2001)
  - PFAS brand names associated with cleaning include Novec (3M) and PolyFox (OMNOVA Solutions)
- Surface coating, paint, and varnish
  - O PFASs have been used in coating, paint, and varnish to reduce surface tension for substrate wetting, levelling, dispersing agents, and improving gloss and antistatic properties. In dyes and inks, they can be used as pigment grinding aids and combat pigment flotation problems. (UNEP 2011) Perfluorinated urethanes enhance the protective properties of anticorrosive paints. (Kissa 2001)
  - o Certain ski waxes use PFAS. (Charonnat 2001) (Plassmann and Berger 2013)
- Plastics, resins, and rubbers
  - PFOA is used to manufacture certain plastics or applied plastics such as polytetrafluoroethylene (PTFE) and polyvinylidine fluoride (PVDF). PTFE has hundreds of uses in consumer and industrial products such as applications noted elsewhere in this paper of textiles, medical industries, cookware, etc. PVDF has unique and useful properties, and so it is used in critical industrial applications like handling chemicals, automotive fuel hoses, electrical cable insulation and jacketing, architectural coatings, high purity piping, and semiconductor piping. (van der Putte 2010)

- o Ammonium perfluorononanoate (APFN CASRN 3658-62-6) is primarily used as a processing aid in fluoropolymer manufacture, most notably polyvinylidene fluoride (PVDF). (Prevedouros et al. 2006)
- o PFASs are used as mold-release agents for thermoplastics, polypropylene, epoxy resins, polyurethane elastomer foam molding. (Kissa 2001)
- PFASs have been used in formulations for antiblocking agents for vulcanized and unvulcanized rubbers. (Kissa 2001)
- o PFASs in silicone rubber sealants make the seal soil resistant. (Kissa 2001)
- PFASs improve wetting of fibers or fillers in composite resins and speed escape of bubbles. (Kissa 2001)
- Perfluorobutanesulfonate (PFBS CASRN 375-73-5) has been used as a flame retardant for polycarbonate by Miteni under the brand name RM 65. (Miteni product information) Other polycarbonates are described as having PTFE in them. (RTP Co. http://web.rtpcompany.com/info/data/0300/flame.htm)
- o PTFE is best known by brand name Teflon (DuPont, now Chemours).

#### Adhesives

- o PFASs are used in solvent-based and water-based adhesives to assure a complete contact between the joining surfaces and retard foaming. PFAS surfactants added to rubber allows adhesiveless bonding to steel. (Kissa 2001)
- PFAS brand names associated with adhesives include Zonyl FSN-100, FSO-100, FSA, FSP, and FSN. (Kissa 2001)

# Antifogging

- o PFASs can be used on glass, metal, or plastic surfaces as a antimist film to prevent fogging of surfaces in humid environments such as bathrooms, automobile windshields, and eyeglass lenses. PFASs can also be used for the same with glass and plastic cover sheets used in agriculture. PFASs can be blended into transparent polyvinyl chloride, polyethylene, or ethylene-vinyl acetate film to reduce clouding. (Kissa 2001)
- O Potassium perfluorooctane sulfonate (CASRN 2795-39-3) and nonionic surfactants are known to have similar uses. (Kissa 2001)

#### Cement additives

- o PFASs reduce shrinkage of cement. (Kissa 2001)
- Cement tiles containing PFAS are more weather resistant than tiles made with other dispersants. (Kissa 2001)
- o PFASs improve primers used for coating cement mortar. (Kissa 2001)

#### Oil industry

- PFASs may be used as surfactants to enhance recovery in oil or gas recovery wells. (UNEP 2011) (Kissa 2001)
- PFASs may be used as evaporation inhibitors for gasoline, and as jet fuel and hydrocarbon solvents. (UNEP 2011)
- PFASs have been used in civil and military hydraulic oils to prevent evaporation, fires, and corrosion. (UNEP 2011)

 Petroleum-product storage tanks may use a floating layer of cereal grains treated with PFAS on top of the liquid surface to reduce evaporation loss. Similarly, evaporation of hydrocarbon fuel can be prevented by an aqueous layer containing PFAS. (Kissa 2001)

# Mining industry

- PFASs may have been used as surfactants to enhance recovery of metals from ores in copper and gold mines. (UNEP 2011)
- o PFASs are used in the ore flotation process. This includes aluminum and vanadium. (Kissa 2001)
- o PFASs are also used in nitrogen flotation to recover uranium. (Kissa 2001)

# Photographic industry

- PFASs have been used in manufacturing film, paper, and plates as dirt rejecters and friction control agents and to reduce surface tension and static electricity. (UNEP 2011) (Kissa 2001)
- PFOA (CASRN 335-67-1) and PFOS (CASRN 1763-23-1) have both been used in this industry. (UNEP 2011; van der Putte 2010)
- o Photography industry users of PFAS include producers of consumer film, X-ray film for medical and industrial use, and the movie industry. (UNEP 2011)

#### Electronics industry

- PFASs are used in the manufacturing of digital cameras, cell phones, printers, scanners, satellite communication systems, radar systems, and more. (UNEP 2011)
- Cured epoxy resins are removed from integrated circuit modules by solutions containing small amounts of PFAS. (Kissa 2001)
- o PFOA is used to make fluoropolymers that are used in cable and wire insulation for computer networks. (van der Putte 2010) Insulated wire may be prepared by coating the wire electrophoretically and treating the wire with PFAS before baking. (Kissa 2001)
- Electric circuits may be sealed with a material that contains PFAS. (Kissa 2001)
- o The products themselves are mostly PFAS-free.
- o Zinc battery electrolyte may contain PFAS. Alkaline manganese batteries may have MnO<sub>2</sub> cathodes treated with PFAS. (Kissa 2001)
- o PFASs are used in low-foaming noncorrosive wetting agents in solders for electrical parts and cleaning of electronic components. (Kissa 2001)
- PFASs are used as lubricants coated on the surface of magnetic recording devices such magnetic tape, floppy disks, and disk drives. (Kissa 2001)

#### • Semiconductor industry

- PFASs are used to reduce surface tension and reflectivity of etching solutions for precise photolithography in the semiconductor industry. (UNEP 2011)
- o They are used in liquid etchant in photo mask rendering process. (UNEP 2011)
- PFOA and PFOS have both been used in this industry. (UNEP 2011; van der Putte 2010)

# Etching

o PFASs are used as wetting agents in etch baths. This includes glass etching, plastics etching, fused silica, and aluminum. They are also used in the semiconductor industry etching as noted above. (Kissa 2001)

# • Cosmetics and personal care

- PFASs are used in cosmetics as emulsifiers, lubricants, or oleophobic agents.
   PFASs are also used in hair-conditioning formulations and hair creams. (Kissa 2001)
- PFASs can be used in toothpaste to increase fluoride-enamel interactions. (Kissa 2001)

#### Pesticides

- N-Ethyl perfluorooctane sulfonamide (EtFOSA; sulfluramid; CASRN 4151-50-2) is an insecticide whose registration was cancelled in May 2008. (EPA-HQ-OPP-2007-1082; FRL-8364-2)
- PFASs may be used as inert surfactants in pesticide products. Potassium N-ethyl-N-[(heptadecafluorooctyl) sulfonyl] glycinate (CASRN 2991-51-7) and 3-[(heptadecafluorooctyl)sulfonyl]amino]-N,N,N-trimethyl 1-propanaminium iodide (CASRN 1652-63-7) have been used in pesticide formulations. (UNEP 2011)
- PFASs can be used as dispersants and wetting agents for herbicides and to aid wetting and penetration in insecticides. (Kissa 2001)

#### Medical uses

- Most video endoscopes contain a small amount of PFAS. (UNEP 2011)
- PFASs are used as a dispersant in radio-opaque ETFE production for accuracy and precision in medical devices such as radio-opaque catheters for angiography and in-dwelling needle catheters. (UNEP 2011)
- o Perfluorodecanaoic acid (PFDA CASRN 335-76-2) grafted onto polyurethane improved its compatibility with blood. (Kissa 2001)
- o PFASs facilitate dispersion of cell aggregates from tissues in a saline solution, used to diagnose cell abnormalities. (Kissa 2001)

#### Oil spills

- Oil spills on water can be contained and prevented from spreading by injecting a chemical barrier containing PFAS into the water. (Kissa 2001)
- Perlite or vermiculite treated with a cationic PFAS is claimed to be helpful in containing oil spills. (Kissa 2001)

Please direct any questions about this document to Linda Gaines in US EPA, Office of Land and Emergency Management, Office of Superfund Remediation and Technology Innovation, at <a href="maintenangement-gaines.linda@epa.gov">gaines.linda@epa.gov</a> or 703-603-7189.

#### Works Cited

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# Harris, Kimberly

From:

Harris, Kimberly

Sent:

Thursday, September 14, 2017 3:51 PM

To:

Pepin, Rob

Subject:

RE: Request for Agenda Items for the September 14 Region 5 State/Tribal WQS Call - 9:30

am - 10:30 am (Central)

Hi Rob.

Let's hold off on that one for now. I tried circling back to the MDEQ folks who months back indicated that they wanted permit info (what other states are doing & national direction). However, focus now seems to have shifted to other PFAS areas (like sources, fate and transport). After today's call, I also received an email from MDEQ requesting assistance on a possible chromium electroplating/WWTP issue.

Thanks!
-Kim



Kimberly M. Harris U.S. EPA-Region 5 PFAS Regional Coordinator

(312) \$55-4235 Work Harris Herberty Steps gov

Water Division/GWDWB 77 W. Jackson Boulevard Chicago, Illinois 60604-3590

From: Pepin, Rob

**Sent:** Thursday, September 14, 2017 11:15 AM **To:** Harris, Kimberly <a href="mailto:kimberly@epa.gov">harris, kimberly@epa.gov</a>

Subject: RE: Request for Agenda Items for the September 14 Region 5 State/Tribal WQS Call - 9:30 am - 10:30

am (Central)

Thanks. Do you still want me to get that 3M permit from Minnesota? I forgot about it.

Rob Pepin

Robert Pepin | NPDES Programs Branch, US Environmental Protection Agency, Region 5 | 77 W Jackson Blvd, WN-16J, Chicago, IL 60604 | ph: (312) 886-1505 | **Pepin.Robert@epa.gov** 

From: Harris, Kimberly

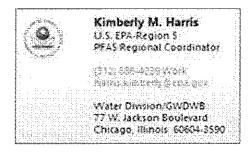
Sent: Thursday, September 14, 2017 09:28 AM

To: Anson, Robie <anson.robie@epa.gov>; Poleck, Thomas <poleck.thomas@epa.gov>;

(Sarah.Yang@wisconsin.gov) <Sarah.Yang@wisconsin.gov>; Tschampa, Andrew <tschampa.andrew@epa.gov>; angela.preimesberger@state.mn.us; Audrey Rush (audrey.rush@epa.ohio.gov) <audrey.rush@epa.ohio.gov>; Koch, Brian <br/>
brian.koch@illinois.gov>; Bauer, Candice <br/>
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Attached are slides for today's PFAS talk.



From: Anson, Robie

Sent: Tuesday, September 12, 2017 1:39 PM

To: Poleck, Thomas <poleck.thomas@epa.gov>; (Sarah.Yang@wisconsin.gov) <Sarah.Yang@wisconsin.gov>; Tschampa, Andrew <tschampa.andrew@epa.gov>; angela.preimesberger@state.mn.us; Audrey Rush (audrey.rush@epa.ohio.gov) <audrey.rush@epa.ohio.gov>; Koch, Brian <bri>spian.koch@illinois.gov>; Bauer, Candice <bauer.candice@epa.gov>; chess@idem.IN.gov; Catherine Laux (claux@kbic-nsn.gov) <claux@kbic-</pre> nsn.gov>; Celeste Hockings (chockings@ldftribe.com) <chockings@ldftribe.com>; chris.skalski@epa.state.oh.us; Pfeifer, David <pfeifer.david@epa.gov>; Dee.allen@ldftribe.com; Dennis Wasley (dennis.wasley@state.mn.us) < dennis.wasley@state.mn.us>; Dobbins, Laura (MPCA) <Laura\_Dobbins@state.mn.us>; dgcox.mitw.org <dgcox@mitw.org>; Edly, Kay <Edly, Kay@epa.gov>; Hammer, Edward <a href="mailto:hammer.edward@epa.gov">hammer, Edward <a href="mailto:hammer.edward@epa.gov">hammer, Edward <a href="mailto:hammer.edward@epa.gov">hammer, Edward <a href="mailto:hammer.edward@epa.gov">hammer.edward@epa.gov</a>; flemik@dnr.state.wi.us; Kevin Goodwin <goodwink@michigan.gov>; Greg Searle <greg.searle@dnr.state.wi.us>; Hack, Eileen <EHack@idem.IN.gov>; Hemken, Meghan <Hemken.Meghan@epa.gov>; Henningsgaard, Bruce (MPCA) <bruce.henningsgaard@state.mn.us>; Jason DeVries (jdevries@ldftribe.com) < jdevries@ldftribe.com>; Jim Schmidt <james.w.schmidt@dnr.state.wi.us>; Johnson, Aaron <Johnson.AaronK@epa.gov>; kkruse@kbic-nsn.gov; Mayo, Kathleen <mayo.kathleen@epa.gov>; Kristi Minahan <Kristi.minahan@wisconsin.gov>; Lathrop, Johanna L - DNR <Johanna.Lathrop@wisconsin.gov>; laura.solem@state.mn.us; linda.nguyen@redcliff-nsn.gov; Margaret Watkins (mwatkins@grandportage.com) < mwatkins@grandportage.com>; Nord, Mari < nord.mari@epa.gov>; Marko, Katharine < Marko. Katharine@epa.gov >; Melis Z. Arik < marik@oneidanation.org >; nancyschuldt@fdlrez.com; Naomi Tillison <WQS@badriver-nsn.gov>; nathan.podany@scc-nsn.gov;

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Cc: Harris, Kimberly < harris.kimberly@epa.gov >

Subject: RE: Request for Agenda Items for the September 14 Region 5 State/Tribal WQS Call - 9:30 am - 10:30 am (Central)

Hi all,

Just a reminder/last call for agenda items and/or specific questions for Kim Harris for Thursday's call.

Please let me know ASAP.

Thank you,

Robie Anson

US EPA Region 5 Water Quality Branch WQ-16J 77 W. Jackson Blvd. Chicago, IL 60604

(312) 886-1502

From: Poleck, Thomas

Sent: Thursday, August 31, 2017 1:47 PM

To: (Sarah.Yang@wisconsin.gov) < Sarah.Yang@wisconsin.gov>; Tschampa, Andrew <tschampa.andrew@epa.gov>; angela.preimesberger@state.mn.us; Anson, Robie <anson.robie@epa.gov>; Audrey Rush (audrey.rush@epa.ohio.gov) <audrey.rush@epa.ohio.gov>; Koch, Brian <bri>brian.koch@illinois.gov>; Bauer, Candice <bauer.candice@epa.gov>; chess@idem.IN.gov; Catherine Laux (claux@kbic-nsn.gov) <claux@kbic-nsn.gov>; Celeste Hockings (chockings@ldftribe.com) <chockings@ldftribe.com>; chris.skalski@epa.state.oh.us; Pfeifer, David <pfeifer.david@epa.gov>; Dee\_allen@ldftribe.com; Dennis Wasley (dennis.wasley@state.mn.us) < dennis.wasley@state.mn.us>; Dobbins, Laura (MPCA) < Laura. Dobbins@state.mn.us>; dgcox.mitw.org < dgcox@mitw.org>; Edly, Kay <<u>Edly.Kay@epa.gov</u>>; Hammer, Edward <<u>hammer.edward@epa.gov</u>>; flemik@dnr.state.wi.us; Kevin Goodwin <goodwink@michigan.gov>; Greg Searle <greg.searle@dnr.state.wi.us>; Hack, Eileen <<u>EHack@idem.IN.gov</u>>; Hemken, Meghan <<u>Hemken.Meghan@epa.gov</u>>; Henningsgaard, Bruce (MPCA) <bruce.henningsgaard@state.mn.us>; Jason DeVries (jdevries@ldftribe.com) < jdevries@ldftribe.com>; Jim Schmidt <james.w.schmidt@dnr.state.wi.us>; Johnson, Aaron <Johnson.AaronK@epa.gov>; kkruse@kbic-nsn.gov; Mayo, Kathleen <mayo.kathleen@epa.gov>; Kristi Minahan <Kristi.minahan@wisconsin.gov>; Lathrop, Johanna L - DNR <Johanna.Lathrop@wisconsin.gov>; laura.solem@state.mn.us; linda.nguyen@redcliff-nsn.gov; Margaret Watkins (mwatkins@grandportage.com) <mwatkins@grandportage.com>; Nord, Mari <nord.mari@epa.goy>; Marko, Katharine <Marko.Katharine@epa.gov>; Melis Z. Arik <marik@oneidanation.org>; nancyschuldt@fdlrez.com; Naomi Tillison < WQS@badriver-nsn.gov >; nathan.podany@scc-nsn.gov; Donnelly, Peggy Ponnelly.Peggy@epa.gov>; Pepin, Rob pepin.robert@epa.gov>; Phil Monson <phil.monson@state.mn.us>; Phillips, Jennifer <phillips.Jennifer@epa.gov>; schmij@dnr.state.wi.us; Scott Knowles - MPCA (scott.knowles@state.mn.us) <scott.knowles@state.mn.us>; Scott Twait (scott.twait@illinois.gov) <scott.twait@illinois.gov>; SBowe@redlakenation.org; Sharon.Gayan@wisconsin.gov; Shelley Warwick <shelley.warwick@wisconsin.gov>; Soo-Hoo, Mimi

<<u>Soo-Hoo.Mimi@epa.gov</u>>; <u>skozich@kbic-nsn.gov</u>; Poleck, Thomas <<u>poleck.thomas@epa.gov</u>>; tina.vanzile.scc-nsn.gov <<u>tina.vanzile@scc-nsn.gov</u>>; William Cole <<u>William.Cole@state.mn.us</u>>
Cc: Harris, Kimberly <<u>harris.kimberly@epa.gov</u>>
Subject: Request for Agenda Items for the September 14 Region 5 State/Tribal WQS Call - 9:30 am - 10:30 am (Central)

Good afternoon.

If you have any items that you would like to discuss during the September call please send an email to Robie Anson, anson.robie@epa.gov. I will be out of the office the next two weeks and will miss this call.

Kim Harris, our Regional perfluorinated chemical coordinator will be joining the call to discuss Regional activities and issues with PFCs. Please also send any specific questions or items that you would like Kim to address to Robie.

Please note the new access number for this call.

Call-in number and access code: : 866-299-3188 / 312 886 0234#

Regional staff meet in Room 1612.

Tom

Tom Poleck | U.S. EPA Region 5, Water Quality Branch 77 W. Jackson Blvd. (Mail Code: WQ-16J), Chicago, IL 60604

312-886-0217 poleck.thomas@epa.gov www.epa.gov



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August 3, 2017

# Via Certified Mail

E.I. du Pont de Nemours and Company c/o CT Corporation System, Registered Agent 160 Mine Lake Court, Suite 200 Raleigh, NC 27615-6417

Ellis H. McGaughy Fayetteville Works Plant Manager The Chemours Company FC, LLC 22828 NC Highway 87 West Fayetteville, NC 28306-7332

V. Anne Heard Region 4 Administrator U.S. Environmental Protection Agency 61 Forsyth Street, S.W. Mail Code: 9T25 Atlanta, GA 30303-8960

Michael S. Regan Secretary N.C. Department of Environmental Quality 1601 Mail Service Center Raleigh, NC 27699-1601

Michael Scott N.C. DEQ, Division of Waste Management 1646 Mail Service Center Raleigh, NC 27699-1646

Josh Stein
N.C. Attorney General
N.C. Department of Justice
9001 Mail Service Center
Raleigh, NC 27699-9001

The Chemours Company FC, LLC c/o CT Corporation System, Registered Agent 160 Mine Lake Court, Suite 200 Raleigh, NC 27615-6417

Scott Pruitt Administrator U.S. Environmental Protection Agency William Jefferson Clinton Building 1200 Pennsylvania Avenue, N. W. Mail Code: 1101A Washington, DC 20460

Jeff Sessions
U.S. Attorney General
U.S. Department of Justice
950 Pennsylvania Avenue, NW
Washington, DC 20530-0001

S. Jay Zimmerman N.C. DEQ, Division of Water Resources 1611 Mail Service Center Raleigh, NC 27699-1611

Governor Roy Cooper N.C. Office of the Governor 20301 Mail Service Center Raleigh, NC 27699-0301 Re: Notice of Intent to Bring Citizen Suit Against:

- (a) The Chemours Company FC, LLC (Chemours) and
- (b) E.I. DuPont de Nemours & Company (DuPont)

#### Ladies and Gentlemen:

Our law firm represents Cape Fear Public Utility Authority ("CFPUA") in connection with perfluorinated chemicals ("PFCs") released by Chemours and DuPont into the Cape Fear River at the Fayetteville Works facility. The Fayetteville Works facility is a chemical manufacturing facility owned and operated by Chemours and is located on the Cape Fear River near Fayetteville, North Carolina. The Cape Fear River is a major source for drinking water.

CFPUA is a water authority created pursuant to North Carolina General Statutes Chapter 162A that provides potable water to residents of New Hanover County and the City of Wilmington. One of CFPUA's water supply intakes is located on the Cape Fear River in Bladen County, North Carolina, downstream of the Fayetteville Works facility.

You are hereby provided notice that CFPUA, through undersigned counsel, intends to file a lawsuit against Chemours and DuPont in federal court to enforce the requirements of the Clean Water Act ("CWA") and Resource Conservation and Recovery Act ("RCRA"). The federal court complaint will allege that: Chemours is in continuing violation of an "effluent standard or limitation" under the CWA (33 U.S.C. § 1365(a)(1)); and Chemours and DuPont have contributed, and Chemours is contributing, "to the past or present handling, storage, treatment, transportation, or disposal of any solid or hazardous waste which may present an imminent and substantial endangerment to health or the environment" in violation of RCRA (42 U.S.C. § 6972(a)(1)(B)). This notice is provided to you pursuant to and in accordance with the requirements of Section 505(b)(1)(A) of the CWA, 33 U.S.C. § 1365(b)(1)(A), and Section 7002(b)(2)(A) of RCRA, 42 U.S.C. § 6972(b)(2)(A).

In accordance with CWA Section 505(b)(1)(A) and applicable rules governing the provision of notice of intent to file a CWA citizen suit at 40 C.F.R. Part 135, and RCRA Section 7002(b)(2)(A) and applicable rules governing the provision of notice of intent to file a RCRA citizen suit at 40 C.F.R. Part 254, CFPUA hereby provides the following information:

#### A. Facts

## 1) Fayetteville Works Facility operations

Chemours was a wholly owned subsidiary of DuPont when it acquired the Fayetteville Works facility from DuPont on February 1, 2015. Chemours later separated from DuPont in July 2015. The Fayetteville Works facility had been operated by DuPont since the early 1970s. DuPont still operates a manufacturing area at the facility.

The Fayetteville Works facility has five discrete manufacturing areas: (i) Fluoromonomers/Nafion (operated by Chemours); (ii) Polymer Processing Aid ("PPA") (operated by Chemours); (iii) Butacite (operated by Kuraray and rented from Chemours); (iv) SentryGlas

(operated by Kuraray and rented from Chemours); and (v) Polyvinyl Fluoride ("PVF") (operated by DuPont and rented from Chemours). The wastewater from each of the manufacturing areas flows through Chemours' on-site wastewater treatment plant ("WWTP"), is diluted with much larger volumes of water, and is ultimately discharged into the Cape Fear River at Outfall 002. The Fayetteville Works facility is operating under NPDES Permit No. NC0003573 (the "NPDES Permit"), the most recent version of which was issued to Chemours for the point source discharge from the entire Fayetteville Works facility.

According to a Chemours representative, the Fayetteville Works facility has been generating and discharging a substance or group of substances, now identified as GenX, as a byproduct since 1980. Based upon information and belief, the generation of GenX at the Fayetteville Works facility, as a product, byproduct or otherwise, has been and is accompanied by the generation of GenX Pollutants which are defined in this notice as follows:

- · chemicals collectively identified by DuPont and Chemours as "GenX";
- chemicals that are structurally or functionally or otherwise similar to GenX that result from or are associated with the manufacture, use, processing, treatment, or disposal of GenX;
- perfluoroalkyl ether carboxylic acids (PFECAs); and
- chemicals that are structurally or functionally or otherwise similar to PFECAs that result from or are associated with the manufacture, use, processing, treatment, or disposal of PFECAs.

More specifically, the GenX Pollutants include, <u>without limitation</u>, the following GenX Pollutants found in the Cape Fear River in the vicinity of the CFPUA intakes:

- GenX (described in more detail in section A.3 of this notice);
- Perfluoroalkyl ether carboxylic acids with one ether group (mono-ether PFECAs) including:
  - o Perfluoro-2-methoxyacetic acid; C<sub>3</sub>HF<sub>5</sub>O<sub>3</sub>; CAS No. 674-13-5
  - o Perfluoro-3-methoxypropanoic acid; C<sub>4</sub>HF<sub>7</sub>O<sub>3</sub>; CAS No. 377-73-1
  - o Perfluoro-4-methoxybutanoic acid; C<sub>5</sub>HF<sub>9</sub>O<sub>3</sub>; CAS No. 863090-89-5
- Perfluoroalkyl ether carboxylic acids with multiple ether group (multi-ether PFECAs) including:
  - o Perfluoro(3,5-dioxahexanoic) acid; C<sub>4</sub>HF<sub>7</sub>O<sub>4</sub>; CAS No. 39492-88-1
  - o Perfluoro(3,5,7-trioxaoctanoic) acid; C<sub>5</sub>HF<sub>9</sub>O<sub>5</sub>; CAS No. 39492-89-2
  - o Perfluoro(3,5,7,9-tetraoxadecanoic) acid; C<sub>6</sub>HF<sub>11</sub>O<sub>6</sub>; CAS No. 39492-90-5

By 2011, in addition to its generation as a byproduct, GenX was intentionally manufactured at the Fayetteville Works facility as a replacement for another substance or group of substances called "C8" (CAS No. 3825-26-1). C8 is also known as perfluorooctanoic acid ("PFOA") or its salt, ammonium perfluorooctanoate ("APFO"). The substance is a long-chain synthetic

perfluorinated carboxylic acid<sup>1</sup> used in the manufacturing of products such as Teflon. CFPUA is informed and believes that C8 was discontinued because of its adverse effects on the environment and human health and that studies indicate GenX has similar adverse effects. Based on information and belief, Chemours and DuPont have released, and Chemours continues to release, GenX and GenX Pollutants into air, soil, surface water, and groundwater at the Fayetteville Works facility.

## 2) DuPont's NPDES Permit applications and production of C8

In or around December 1995, DuPont submitted to North Carolina Department of Environment and Natural Resources<sup>2</sup> ("DENR"), as part of its NPDES Permit renewal application, a request to reroute the Nafion wastewater to bypass the facility's wastewater treatment plant. According to DuPont, the only significant pollutant in the "low biodegradable" wastewater was fluoride, which was not removed in the water treatment process, so the wastewater only added to the hydraulic load at the WWTP. The 1996 NPDES Permit appears to have authorized the bypass. Based upon information and belief, the Nafion wastewater also included GenX Pollutants.

On May 3, 2001, DuPont submitted a renewal application for its 1996 NPDES Permit in which the company disclosed its intent to begin manufacturing C8 at the Fayetteville Works facility. DuPont had previously been purchasing C8 from 3M, but CFPUA is informed and believes that 3M stopped manufacturing the substance due to concerns over its persistence, bioaccumulation and toxicity. CFPUA is informed and believes that, by the time of its 2001 NPDES renewal application:

- a) DuPont had been conducting medical studies on C8 for decades. DuPont already "understood that PFOA [i.e., C8] caused cancerous testicular, pancreatic, and liver tumors in lab animals. One laboratory study suggested possible DNA damage from PFOA exposure, and a study of workers linked exposure with prostate cancer." Nathaniel Rich, The Lawyer Who Became DuPont's Worst Nightmare, The NY Times Magazine, Jan. 6, 2016.
- b) In 1991, DuPont set an internal exposure guideline of 1 ppb.
- c) DuPont had been the defendant in a federal lawsuit over adverse health effects arising from C8 contamination from its facility in Parkersburg, West Virginia, and a class action regarding adverse health effects was filed against the company in August 2001.

However, CFPUA is informed and believes that DuPont in its 2001 NPDES Permit renewal application failed to disclose any of the studies or health data on C8 in its possession. Instead, DuPont represented to DEQ's Division of Water Quality<sup>3</sup> ("DWQ") that: (i) based on "medical surveillance of its own employees and epidemiological data from others in the industry," C8 "does not pose a health concern to humans or animals at levels present in the workplace or environment"; (ii) DuPont had used C8 for forty years "with no observed health effects in workers"; and (iii) the compound "is neither a known developmental toxin nor a known human carcinogen." The 2001

<sup>&</sup>lt;sup>1</sup> PFOA is considered a long-chain PFC because it has a string of eight carbons off the carboxylic acid compound—hence the name "C8."

<sup>&</sup>lt;sup>2</sup> Now Department of Environmental Quality ("DEQ").

<sup>&</sup>lt;sup>3</sup> Now Division of Water Resources ("DWR").

NPDES Permit application requested authorization to discharge the C8 wastewater directly to a dedicated outfall, bypassing the facility's WWTP. CFPUA understands that, beginning in the fall of 2002, DuPont started manufacturing C8 at the Fayetteville Works facility. The renewal NPDES Permit, however, was not issued until January 2004. Because later submissions from DuPont represented that the C8 manufacturing operation was constructed to have no process wastewater discharges, and that the wastewater would be captured and incinerated off site, the 2004 NPDES Permit did not include authorization for discharge of the C8 manufacturing wastewater. Upon information and belief, the wastewater from C8 manufacturing included GenX Pollutants.

# 3) Phase out of C8 and transition to GenX

In 2006, EPA initiated a voluntary PFOA Stewardship Program, in which DuPont participated, calling for the complete elimination of PFOA (*i.e.*, C8) both from emissions to all media and from product content by 2015. DuPont identified GenX as a viable replacement compound. GenX (CAS Nos. 13252-13-6 and 62037-80-3) falls within a category of chemicals known as short-chain<sup>4</sup> PFECAs.

DuPont submitted its next NPDES Permit renewal application on May 1, 2006. As to the manufacture of C8, DuPont represented in its application that: (i) the wastewater "is collected and shipped off-site for disposal"; (ii) "[n]o process wastewater from this manufacturing facility is discharged to the site's biological WWTP or to the Cape Fear River"; and (iii) the C8 produced at the facility "is used to produce fluoropolymers and fluorinated telomers, but none of the produced [C8] is used at the Fayetteville Works site." As to the Nafion manufacturing operations, DuPont disclosed in its application that the plant manufactures five final products, including FLPR Vinyl Ether monomers and HFPO monomers (hexafluoropropylene oxide, CAS No. 428-59-1). According to DuPont, the Vinyl Ether and HFPO monomers are shipped to other DuPont locations to produce various fluorochemical products such as Teflon, and the Nafion wastewater was now being treated in the facility's WWTP.

The renewal NPDES Permit was issued May 25, 2007. Although all C8 process wastewater was to be captured and disposed of off-site, the permit included a monitoring condition for C8 due to known groundwater contamination.

In 2008, DuPont submitted to the EPA pre-manufacture notices for GenX (P-08-508 and P-08-509) pursuant to the Toxic Substances Control Act ("TSCA"). On January 28, 2009, EPA and DuPont entered the TSCA Consent Order governing the manufacture of GenX. The Consent Order specified that "EPA has concerns that [GenX] will persist in the environment, could bioaccumulate, and be toxic ('PBT') to people, wild mammals, and birds," and that, based on available data, "EPA has human health concerns" for GenX. TSCA Consent Order at vii. Due to the likelihood that GenX would be used as a substitute for C8, EPA determined that "more information is needed on the toxicity and pharmacokinetics" of GenX, and noted the "high concern for possible environmental effects over the long-term." TSCA Consent Order at xi–xii. Accordingly, EPA concluded that "uncontrolled manufacture, import, processing, distribution in

<sup>&</sup>lt;sup>4</sup> GenX is a short-chain PFC in that two shorter carbon chains are connected by an ether linkage as opposed to the unbroken eight-carbon chain in C8.

commerce, use, and disposal of [GenX] may present an unreasonable risk of injury to human health and the environment." TSCA Consent Order at xv. Due to the stated concerns of EPA, the Consent Order authorized the manufacture of GenX but required that DuPont "recover and capture (destroy) or recycle [GenX] at an overall efficiency of 99% from all effluent process streams and the air emissions (point source and fugitive)." TSCA Consent Order at 36.

As part of its NPDES permit renewal process, DuPont representatives, including its environmental manager Michael Johnson, met in August 2010 with DWQ to discuss the phase-out of C8. During that meeting, CFPUA is informed and believes that DuPont identified the C8 replacement as "GenX" and, consistent with the disclosures in its renewal application, represented that the wastewater generated from the manufacture of GenX would be captured and disposed of off-site.

On April 29, 2011, DuPont submitted another NPDES Permit renewal application. CFPUA is informed and believes that DuPont had begun transitioning from C8 to GenX by that time. Where its disclosures previously identified the manufacture of C8, DuPont instead identified the manufacturing area as a "PPA [polymer processing aid] manufacturing area." DuPont represented in its application that: (i) the "processing aids produced in this unit are used to produce fluoropolymers and fluorinated telomers, but none of the produced processing aids are used at the Fayetteville Works site"; (ii) "[a]ll process wastewater generated from this manufacturing facility is collected and shipped off-site for disposal"; and (iii) "[n]o process wastewater from this manufacturing facility is discharged to the site's biological WWTP or to the Cape Fear River." DuPont's representations regarding the Nafion plant are essentially identical to its May 2006 NPDES application. The effluent from the Nafion wastewater is represented as being heavily diluted with noncontact river water and other water prior to discharge.

The NPDES Permit renewal was issued February 6, 2012, and advised DuPont that the Cape Fear River segment into which DuPont is discharging wastewater had been reclassified to a water supply classification—WS-IV. As with the prior NPDES permit, PFOA (i.e., C8) monitoring conditions were included; PFOA monitoring was required at Outfall 002—after dilution with large volumes of non-contact river water and other water. The 2012 NPDES Permit does not authorize the discharge of GenX or any other GenX Pollutants.

## 4) RCRA investigation of C8 contamination

At some point after DuPont began the manufacture of C8 at its Fayetteville Works facility, the site became contaminated with C8 in the soil and groundwater, due (on information and belief) to some combination of spills, leaks, releases, discharges, and air emissions. DuPont conducted a RCRA Facility Investigation (RFI) under the Hazardous and Solid Waste Amendments Corrective Action Program, which led to the issuance of a Phase III RFI Report in February 2014, revised August 2014. According to the RFI Report, at least seven releases occurred between March 2011 and February 2013, including a release from the PPA facility in June 2011, a release from the Nafion facility in March 2012, and a release from the Waste Fluorocarbon Storage Tank in March 2012. Based upon information and belief, at the time of some or all of those releases, DuPont was manufacturing or otherwise producing GenX Pollutants, which are likely to have been contaminants in one or more of the releases.

Earlier RFI reports—in particular the Phase I RFI dated April 14, 2003 and revised August 1, 2003, the Phase II RFI dated June 2006 and its August 2009 Addendum—include additional findings regarding historical contamination and releases at the Fayetteville Works facility. Among other things, the RFIs: (i) identify C8 contamination in soil and groundwater throughout the Fayetteville Works facility, and posit that some of the contamination is due to deposition of C8 air emissions; (ii) indicate that until 1990, unlined lagoons constructed in or around 1979 were used as biosludge settlement lagoons for wastewater from throughout the facility, before discharging to the Cape Fear River; and (iii) acknowledge historical releases at the Nafion manufacturing area, including from solid waste management units (SWMUs) handling Nafion wastewater. CFPUA is informed and believes that DuPont was generating GenX Pollutants at the Fayetteville Works facility during the time of the activities and releases at the facility identified in the RFIs, such that GenX Pollutants were constituents of the contamination and releases described in the RFIs.

# 5) Change of ownership and 2016 NPDES Permit application

On June 19, 2015, DuPont submitted an ownership change request, notifying DWR of the pending transfer of the Fayetteville Works facility to Chemours and requesting a permit amendment. On June 24, 2015, Michael Johnson, Chemours' (and previously DuPont's) environmental manager, met with DWQ officials to discuss a "new" perfluorinated compound identified in the Cape Fear River, GenX, which had been identified by N.C. State University researchers conducting sampling on the Cape Fear River as part of a study commissioned by EPA. The researchers have since published their results, Legacy and Emerging Perfluoroalkyl Substances are Important Drinking Water Contaminants in the Cape Fear River Watershed of North Carolina, in Environmental Science & Technology Letters (November 10, 2016) ("Knappe Report"). CFPUA is informed and believes that DuPont represented to DWQ that GenX was C8's replacement, and that GenX was no longer being discharged to the Cape Fear. The 2012 NPDES Permit was amended to reflect the change of ownership effective July 1, 2015.

Chemours submitted its most recent NPDES Permit renewal application on April 27, 2016. The application contained essentially identical representations regarding the PPA and Nafion manufacturing areas as the April 2011 renewal application. Similar to the prior application, the effluent from the Nafion wastewater is represented as being heavily diluted with noncontact river water and other water prior to discharge.

The April 2016 NPDES renewal application requested that the C8 monitoring condition be removed from the NPDES Permit. Chemours' justification for the request was that C8 monitoring at Outfall 002 showed an average C8 concentration of .027 ug/L—below the recommended Interim Maximum Allowable Concentration ("IMAC") of 1 ug/L. Chemours' further asserted that non-contact water from the Cape Fear River—which Chemours represented constituted 99% of the effluent flow at Outfall 002—had an average C8 concentration of .012 ug/L, thus contributing to the total C8 in the effluent. Based on Chemours' representations: (i) the pre-dilution effluent—only 1% of the final discharge—would be the cause of the increase in C8 concentration from .012 ug/L to .027 ug/L at Outfall 002, and (ii) the actual concentration of C8 in Chemours' effluent prior to dilution by water from the Cape Fear River would be 1.5 ug/L, which exceeds by 50 percent the IMAC upon which Chemours chose to base its request. In addition, groundwater monitoring results in the RFI Report suggest that Chemours' on-site C8 contamination may have

impacted Willis Creek, which flows into Cape Fear River just upstream of Chemours' intake, potentially contributing to the reported 0.12 ug/L concentration of C8 in the Cape Fear River at the Chemours intake.

#### 6) Discovery of GenX in the Cape Fear River

The Knappe Report shows that GenX, along with six other GenX Pollutants, are present in the Cape Fear River, downstream of the Fayetteville Works facility. The report found that GenX was in the raw water at CFPUA's intake at mean concentrations of 631 ng/L, and six other GenX Pollutants (all PFECAs) were found at even higher levels. Based upon information and belief, the Fayetteville Works facility is the source of each of the GenX Pollutants at CFPUA's intake.

The Knappe Report indicates that CFPUA's water treatment plant is largely ineffective at removing PFECAs from the water during the treatment process. With regard to treatment options, although reverse osmosis might be effective, it is not practicable to implement at the scale required given the volume of water treated by CFPUA. Finally, carbon filtration is not known to be effective at removing PFECAs.

Once publicly confronted about GenX contamination in the Cape Fear River, Chemours: (a) asserted that GenX is an "unregulated" chemical that was being discharged as a byproduct from its Nafion-related manufacturing processes, in particular during the manufacture of vinyl ethers; (b) disclosed that GenX was in its air emissions as part of those same processes; and (c) explained that GenX had been discharged from its vinyl ether process, unabated, from 1980 until November 2013, at which time abatement technology was implemented to reduce the GenX discharge by 80 percent. Following public outcry, Chemours committed to taking steps to prevent discharge of any wastewaters containing GenX by June 21, 2017. However, as recently as July 12, 2017, GenX was identified in Chemours' effluent being discharged to the Cape Fear River.

After reviewing the available data regarding the PBT characteristics of GenX and consultation with EPA, North Carolina Department of Health and Human Services ("DHHS") issued a Risk Assessment on July 14, 2017 setting a health goal of 140 ng/L. Subsequent testing has shown GenX present in the raw CFPUA's intake, and in the Authority's finished water, at concentrations in excess of the health goal.

# B. Chemours is violating CWA and RCRA, and Chemours and DuPont have violated RCRA

## 1) The specific standard, limitation or order alleged to be violated under CWA

Section 505(a)(1) of the CWA permits citizens to commence a civil action against "any person... who is alleged to be in violation of... an effluent standard or limitation under this Act," 33 U.S.C. § 1365(a)(1), including the prohibition against discharging pollutants without an NPDES permit. 33 U.S.C. § 1311(a).

"Pollutants" under the CWA include "chemical wastes . . . and industrial . . . waste discharged into water." 33 U.S.C. § 1362(6). GenX Pollutants, including GenX, are "pollutants"

as defined under the CWA. Based on information and belief, GenX Pollutants have been and continue to be among CWA pollutants that are discharged into the Cape Fear River from the Fayetteville Works. Based on information and belief, GenX Pollutants are discharged by DuPont from its manufacturing area to the Cape Fear River, using the Chemours WWTP as a conduit. Based on information and belief, the WWTP does not effectively remove GenX Pollutants from wastewater that is routed through it.

The NPDES Permit does not authorize, and has never authorized, discharge of GenX Pollutants from the Fayetteville Works facility. In order to operate in compliance with an NPDES permit, the permit holder must (1) comply with the express terms of the permit; and (2) not make a discharge of pollutants that was not within the reasonable contemplation of the permitting authority at the time the permit was granted. *Piney Run Pres. Ass'n v. County Comm'rs of Carroll County*, 268 F.3d 255 (4th Cir. 2001). Because Chemours and DuPont never identified the GenX Pollutants as constituents in their effluent, the substances could not have been within the contemplation of DEQ, and are therefore not authorized to be discharged by the NPDES Permit.

In addition, DuPont's and Chemours' unauthorized discharges of GenX Pollutants are causing violations of North Carolina water quality standards adopted and enforced pursuant to the CWA, including water quality standards for "oils, deleterious substances, colored, or other wastes," 15A NCAC 2B .0211(12), and for "toxic substances." 15A NCAC 2B .0208, 15A NCAC 2B .0211 (incorporating 15A NCAC 2B .0208 by reference), and 15A NCAC 2B .0216(3)(a) and (h).

2) The specific permit, standard, regulation, condition, requirement, or order which has allegedly been violated under RCRA

Section 7002(a)(1)(B) of RCRA permits citizens to commence a civil action against:

any person . . . including any past or present generator, past or present transporter, or past or present owner or operator of a treatment, storage, or disposal facility, who has contributed or who is contributing to the past or present handling, storage, treatment, transportation, or disposal of any solid or hazardous waste which may present an imminent and substantial endangerment to health or the environment.

# 42 U.S.C. § 6972(a)(1)(B).

Chemours and DuPont are both in violation of this provision, based on their discharges, disposals and other releases of GenX Pollutants described above and discussed in more detail below.

#### 3) Activities alleged to be a violation of CWA and RCRA

The CWA violations by DuPont and Chemours are the historic and ongoing unpermitted discharges of GenX Pollutants at and from the Fayetteville Works facility (including without limitation discharges from the WWTP) and the resulting violations of water quality standards as previously described.

The RCRA violations by Chemours and DuPont are the historic and ongoing contribution to the disposals and other releases of GenX Pollutants to soil, groundwater, surface water, and air at and from the Fayetteville Works facility, which have contaminated and are contaminating the Cape Fear River. As determined by DHHS in its Risk Assessment, and consistent with the health concerns identified by EPA in its TSCA Consent Order, and as indicated in study reports such as Evaluation of substances used in the GenX technology by Chemours, Dordecht, National Institute for Public Health and the Environment, The Netherlands (2016) at page 3 of 92 (GenX substances "are perfluorinated hydrocarbons and poorly degradable in the environment... [and] are causing similar harmful effects as PFOA (such as carcinogenic [effects] and effects on the liver.")), the GenX released in the Cape Fear River may present an imminent and substantial endangerment to health or the environment. Other GenX Pollutants in the Cape Fear River are similar substances to GenX and should be expected to have similar effects and therefore may present an imminent and substantial endangerment to health or the environment.

The dates of the violations are as identified in Part A above.

On June 27, 2017, CFPUA sent written Requests for Information to Chemours, requesting information regarding, among other things, Chemours' and DuPont's historical use, discharges, releases, and emissions of GenX Pollutants, as well as information regarding GenX Pollutants' effect on human or environmental health. To date, Chemours has refused to provide the requested information. CFPUA expressly reserves the right to supplement its prospective citizen suit with any information currently withheld by Chemours that is later disclosed in discovery.

# 4) Persons responsible for the alleged violation

The persons responsible for the alleged violation currently known to CFPUA include Ellis H. McGaughy, Fayetteville Works plant manager, and Michael E. Johnson, Fayetteville Works plant environmental manager.

#### 5) Name, address, and telephone number of person giving notice

The name and contact information of the person giving notice is as follows:

Cape Fear Public Utility Authority 235 Government Center Drive Wilmington, NC 28403 Attn: Jim Flechtner Telephone: (910) 332-6550

The counsel for CFPUA is Brooks, Pierce, McLendon, Humphrey, and Leonard, L.L.P., of Greensboro, North Carolina. The name and contact information of counsel for the person giving notice is as follows:

George W. House V. Randall Tinsley Joseph A. Ponzi Brooks, Pierce, McLendon, Humphrey & Leonard, L.L.P. 2000 Renaissance Plaza 230 North Elm Street Greensboro, NC 27401 Telephone: (336) 373-8850

Telephone: (336) 373-8850 Facsimile: (336) 378-1001

Messrs. House, Tinsley, and Ponzi are providing this notice as counsel for CFPUA.

# 6) Notice of intent to sue

CFPUA intends to file suit not earlier than 60 days after this notice is given as authorized by the Clean Water Act. CFPUA intends to file suit not earlier than 90 days after this notice is given as authorized by the Resource Conservation Recovery Act.

Pursuant to the CWA, RCRA, and EPA's rules at 40 C.F.R. Part 135 and Part 254, copies of this notice letter are being served on the EPA Administrator, the EPA Regional Administrator, the United States Attorney General, the N.C. DEQ Secretary, the N.C. DWR Director, and the N.C. DWM Director.

Sincerely,

George W. House

V. Randall Tinsley

Joseph A. Ponzi

cc: Bill Lane, NC DEQ

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